

# PATENT SPECIFICATION

NO DRAWINGS

975,587

975,587



Date of Application and filing Complete Specification Oct. 5, 1962.

No. 37817/61.

Application made in Germany (No. C25213 IVa/84c) on Oct. 6, 1961.

Application made in Germany (No. C26687 IVa/84c) on April 7, 1962.

Application made in Germany (No. C27704 V/37a) on Aug. 14, 1962.

Complete Specification Published Nov. 18, 1964.

© Crown Copyright 1964.

Index at acceptance—C3 P(8A, 8D2B2, 8P1C, 8P1D, 8P1E1, 8P1F, 8P4C, 8P5, 8P6X); E1 G62

International Classification:—C 08 f (E 01 c)

## COMPLETE SPECIFICATION

### A Composition for Covering or Filling Cavities and Sealing Joints

We, CHEMISCHE FABRIK GRUNAU G.m.b.H. of 9, Weissfrauenstrasse, Frankfurt, Main, Germany, a body corporate organised under the laws of Germany, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

The invention relates to a self-hardening agent which is intended for immediate use and which is suitable for covering and filling cavities, holes and cracks in a substratum. Examples of the substratum to which the composition may be applied are mixtures of sand, shingle or gravel with hydraulically-set structural materials, for example mortar and concrete, such as are used for making roads, runways or factory floors. The substratum can also be natural stone.

Such mixtures can also be used to seal or fill joints of all types, such as movement and expansion joints of roadways and runways. Such sealing compositions have the advantage that they can satisfactorily be shaped, and are resistant to ageing and to fluctuations in temperature so that they do not breakup with changes in weather and have a very good adhesion after hardening. The agent consists of a mixture of monomeric and polymeric methyl methacrylate, an inorganic filler, such as sand, shingle or gravel and a catalyst for the polymerisation of methyl methacrylate. The surface area of the filler is from 30 to 100 m<sup>2</sup> per kilogram of polymerisable mixture.

The mixture of monomeric and polymeric methyl methacrylate can be obtained by partially polymerising monomeric methyl methacrylate, stopping the polymerisation when enough polymer has formed. The degree of polymerisation can easily be controlled by quenching polymerisation when a suitable viscosity is reached. The mixture can also be

made by dissolving polymeric methyl methacrylate in the monomer. The mixture should preferably contain 25 to 65 parts by weight of polymeric methyl methacrylate to 100 parts by weight of monomer. Such mixtures have usually the most suitable viscosity.

It is advisable to use quartz sand as the inorganic filler, containing as little moisture as possible. For special purposes, the fillers can also contain or consist of glass fibres, can also contain or consist of glass fibres, Coloured fillers, such as black iron oxide, titanium dioxide and heavy spar can also be employed, alone or mixed with sand. The quantity of filler can be varied within comparatively wide limits. Usually, 100 to 400 parts by weight of the inorganic filler are added to 100 parts by weight of polymerisable mixture.

100 to 300 parts by weight of filler are preferably used to 100 parts by weight of polymerisable mixture for joint-filling compositions, but this quantity depends upon the joint and the method of working. With very narrow joints, a comparatively-thin sealing composition must be used, but with wider joints, the composition can be more viscous, i.e. contain more fillers.

Not only the quantity but also the surface area of the filler is important. If a filler with too large a total surface area is used, the mixture of polymeric and monomeric methyl methacrylate will, sometimes, not be completely cured, and, in certain circumstances, may remain completely soft. If the filler has too small a surface, very high reaction temperatures may be reached the resin may shrink and bubbles or cracks can be formed on hardening. The total surface of the fillers should be not more than 100 m<sup>2</sup> and not less than 30 m<sup>2</sup> for each kilogram of polymerisable mixture.

For certain purposes, especially if the mixture is to be applied as a thin layer with

The thickly liquid mixture could easily be poured into the holes, cavities and cracks and cured in 30 minutes to 1½ hours. After curing, a coating-composition was made by mixing

- 5 30.0 parts by weight of a mixture of monomeric methyl methacrylate with 30% by weight of polymeric methacrylate, 0.15 part
- 15 16.0 parts by weight of quartz sand with a grain size of 0 to 0.2 mm.  
 57.0 " " " " " " " " " " 0.06 to 0.4 mm. and  
 32.0 " " " " " " " " " " 0.004 to 0.6 mm.

were incorporated. The pasty mixture was poured on to the surface of the concrete slabs and distributed. Before it was cured some coarse sand was scattered on it to give a rough surface.

The coating was cured after 1 to 1½ hours, and traffic could travel over the surface immediately.

#### EXAMPLE 2

- A joint-sealing composition was made by mixing 100 parts by weight of a syrup containing 70% by weight of monomeric methyl methacrylate and 30% by weight of polymeric methyl methacrylate with 0.5 part by weight of hard paraffin wax, 2.5 parts by weight of a 10% by weight solution of dimethyl-*p*-toluidine in monomeric methyl methacrylate, 43 parts by weight of industrial Pineoil and a paste made from 1.5 parts by weight of dibenzoyl peroxide and 1.5 parts by weight of dibutyl phthalate. 155 parts by weight of talc having a surface area of 80 m<sup>2</sup> per kilogram of polymerisable mixture were incorporated into this mixture.

- The composition was cast to form prisms in accordance with DIN 1164 and left to harden for 5 days at 20°. On being compressed, a working load strength of 10 to 30 kg/cm<sup>2</sup> was found. The test elements with a thickness of 40 mm. could be compressed to a thickness of 28 mm. without any cracks forming. On removing the pressure, the test elements assumed their original shape within 12 hours. In the bending tensile strength test, the test elements were bent without breaking. After being subjected to the action of the bending load, the elements had a bending angle of about 60°, which was reduced to 10° about 12 hours after the load was removed.

#### EXAMPLE 3

- If the procedure according to Example 2 is used, but with 60 parts by weight of industrial Pineoil to 100 parts by weight of the methyl methacrylate mixture, much softer and more plastic elements are obtained. It was no longer possible to test the bending tensile strength by the method of DIN 1164.

#### EXAMPLE 4

- 100 parts by weight of a mixture of monomeric methyl methacrylate with 30% by

weight of hard paraffin wax, 6.0 parts by weight of wood turpentine oil and 0.8 part by weight of a 10% by weight solution of dimethyl-*p*-toluidine in methyl methacrylate. Finally a solution of 0.4 part by weight of dibenzoyl peroxide in 0.4 part by weight of dibutylphthalate was added. Then

weight of polymeric methyl methacrylate were mixed with 0.5 part by weight of hard paraffin wax and 0.25 part by weight of a 10% by weight solution of dimethyl-*p*-toluidine in methyl methacrylate. To this was added a paste of

- 1.5 parts by weight of dibenzoylperoxide in  
 1.5 parts by weight of dibutylphthalate, and  
 300 parts by weight of quartz sand with a grain size of 0.1 to 0.3 mm. were incorporated into the mixture.

The mixture was placed in moulds. After 5 days, the bending tensile strength and the compressive strength were determined. The bending tensile strength was 324 kg./cm.<sup>2</sup> and the compressive strength was 1080 kg./cm.<sup>2</sup>.

#### EXAMPLE 5

When the procedure of Example 4 was followed, replacing the quartz sand by 700 parts by weight of feldspar sand with a surface area of 50 m<sup>2</sup> per kilogram of polymerisable mixture, elements which have a bending tensile strength of 226 kg./cm.<sup>2</sup> and a compressive strength of 923 kg./cm.<sup>2</sup> were obtained.

#### WHAT WE CLAIM IS:—

1. A self-hardening agent intended for immediate use for sealing or filling joints for covering or filling cavities, holes and cracks in a substratum of hydraulically-set structural materials mixed with sand, shingle, gravel or natural stone, which comprises a mixture of monomeric and polymeric methyl methacrylate, a catalyst for the polymerisation of methyl methacrylate and an inorganic filler, the surface area of the filler being from 30 to 100 m.<sup>2</sup> per kilogram of polymerisable mixture.

2. An agent as claimed in claim 1 which contains 25 to 65 parts by weight of polymeric methyl methacrylate to 100 parts by weight of monomeric methyl methacrylate.

3. An agent as claimed in claim 1 and 2 in which the catalyst, preferably an organic diperoxide or hydroperoxide, is mixed with a plasticiser or solvent of high boiling point.

4. An agent as claimed in claims 1 to 3 which contains also a substance for lowering the viscosity of the mixture.

5. An agent as claimed in claim 1 to 4